

Slip bands and Knoop hardness anisotropy in hydrothermal grown ZnO crystals

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Introduction: Zinc oxide (ZnO) is an hexagonal crystal of the wurtzite-type structure with F43m space group. It is a wide band-gap semiconductor with an energy gap of 3.37eV with potential for applications as emitter devices in the blue to ultraviolet region and as a substrate material for GaN based devices. Recently, compact laser ZnO laser has been demonstrated. Single crystals of ZnO have been grown using vapor growth, hydrothermal growth, flux growth and more recently from skull melting methods. Among these hydrothermal and vapor growth are used very commonly practiced. In spite of the availability of ZnO crystals obtained from different methods, structurally high quality crystals are not very common. In this report we present results of synchrotron x-ray topography of hydrothermally growth ZnO crystals to characterize defects and reveal the suitability as a substrate for epitaxy.

Methods and Materials: Hydrothermal autoclaves made of high strength steel were used for crystal growth, with a sealed platinum liner to isolate the crystal growth environment from the walls of the autoclave. Crystals were grown at was at 355°C with a temperature gradient of 10°C declining towards the seed zone. Growth rates on the (0001) basal plane seed plates averaged 10 mils per day for the 30 day runs, but the growth is anisotropic: the ratio of growth rates between the fast (C^+) and slow (C^-) growth directions was 3 to 1. It grows with (0001) and (000 $\bar{1}$) monohedra, (1010) prism and (1011) pyramid face. Different slices of ZnO have been cut from bulk using a wire saw and lapped to thickness ranging from 200 to 300 micron. The final polishing was done on soft polishing pad using 0.03 micron alumina. Crystal (0001) crystal plates were used for synchrotron white beam x-ray topographic characterization. The crystals were aligned using back reflection Laue method. Different reflections were selected by appropriately satisfying for Bragg condition by tilting the crystal with respect to the beam. Complete image of the x-ray topographs were recorded using white beam synchrotron radiation by scanning in the vertical direction. Knoop hardness was studied on c-cut plate at the interval 10° using the indenter.

Results: Fig 1 shows slip bands observed on c-cut plate. No growth dislocations were observed. Possibly the growth dislocations were propagating almost normal to the 'c' face. Also inclusions present in the grown crystal were observed. Possibly the differential thermal expansion between the ZnO crystal and the inclusions was the source for these slip bands. These bands were observed parallel to the six prism faces. The slip band perpendicular the diffraction vector invariably extinguished. The generation of the slip bands suggest that the ZnO single crystal are soft. The Knoop hardness value (Figure 2) oscillates between minimum and maximum with interval of 60°. The hardness value falls to the lowest whenever the longer diagonal of the Knoop indenter is parallel to the prism faces.

Conclusions: The synchrotron white beam x-ray topography study clearly shows that the operating slip systems are predominantly {10-10}. This is in correlation with the Knoop hardness anisotropy.

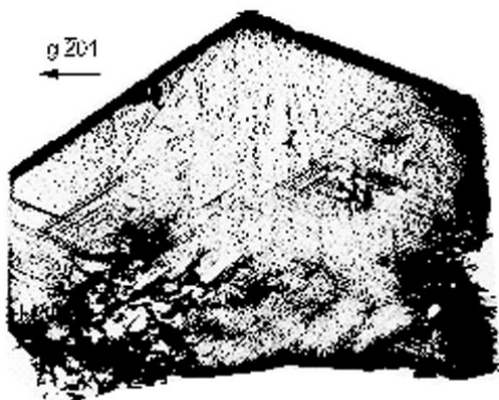


Figure 1. Back reflection x-ray topograph showing slip bands

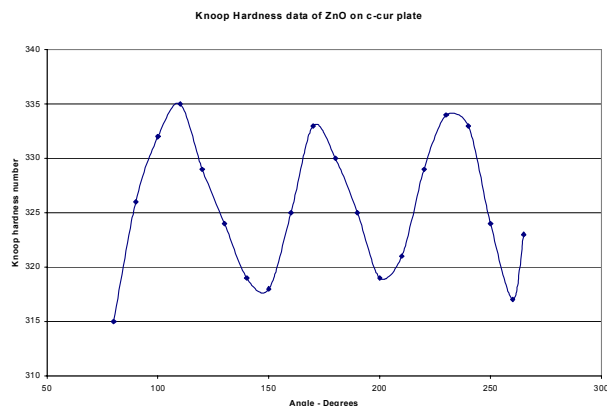


Figure 2. Knoop hardness on (0001) plane